PATENT SPECIFICATION

966,828

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Date of Application and filing Complete Specification: February 4, 1963. *No. 4513/63*

Complete Specification Published: August 19, 1964.

Index at Acceptance:—G1 S (1K, 1T, 4G).

International Classification:—G 01 n.

COMPLETE SPECIFICATION

DRAWINGS ATTACHED

Improvements in or relating to Pour Point Instruments

WE, STANDARD OIL COMPANY, a Corporation organised and existing under the Laws of the State of Indiana, United States of America, of 910 South Michigan Avenue, Chicago 80, State of Illinois, United States of America, do hereby declare the invention, for which we pray that a patent may be granted to us, and the method by which it is to be performed, to be particularly described in and by the following statement:—

This invention relates to an apparatus for determining the pour point of a liquid sample and particularly the pour point of petroleum products.

In the production of various petroleum products one of the most critical production specifications is that of pour point. Pour point is defined as being the lowest temperature at which an oil will just pour, or flow, under its own weight under specified conditions. The test is most generally carried out in the laboratory according to ASTM "Standard Method of Test for Cloud and Pour Points," D 97-57, by placing a small quantity of oil into a standard bottle which is then placed in a succession of freezing mixtures. A thermometer is fixed in the oil, and as the temperature falls, the bottle is removed from the cooling bath at every 5°F. fall in temperature and tilted to determine whether the oil will flow. The lowest temperature at which the oil still remains liquid is known as the pour point. This test is very time consuming, requires the constant attention of an operator and is subject to errors by the operator. The production of many petroleum products such as lubricants and furnace oils is frequently limited by pour point specifications. In such a case a rapid means of measuring the pour point will enable the specification limit to be more nearly approached, and will aid in maximizing the efficiency of the production operation.

According to the invention there is provided apparatus for determining the pour point of a liquid sample, which apparatus comprises a container for said sample, means for cooling said sample to its pour point, means for rocking said container during the cooling of said sample so that the surface of said liquid sample remains in a substantially horizontal plane but so that said surface tilts from the horizontal when the temperature of said sample is at or below its pour point, means for detecting movement of the surface of the sample from a substantially horizontal plane, and means for indicating the temperature of said sample when the surface of said sample moves from a substantially horizontal plane. Preferably the means for detecting movement of the surface of the sample from a substantially horizontal plane comprises a source of a beam of electromagnetic radiation and a sensitive receiver positioned so as to receive the beam after reflection from the substantially horizontal surface of the sample.

The electromagnetic radiation may conveniently be light. There may also be provided a defroster to warm the cooling means, which enables experiments to be carried out in more rapid succession.

The invention also includes a method of determining the pour point of a liquid sample which method comprises cooling the sample and concurrently rocking it in such a way that the surface of the liquid sample remains in a substantially horizontal plane, and then detecting the movement of the sample surface from a substantially horizontal plane and measuring the temperature of the sample at that moment. The movement of the sample surface from a substantially horizontal plane may be arranged to cause a corres-

[Price 4s. 6d.]

ponding movement of the reflection of a beam of electromagnetic radiation issuing from a stationary source and reflected from the sample surface, which movement of the beam is detected by a sensitive receiver. The amplitude and frequency of rocking should preferably be such that no substantial agitation of the surface takes place.

Following is a description by way of example of an embodiment of apparatus in accordance with the invention, reference being made to the accompanying drawings in which :—

Figure 1 is a schematic representation of one form of the pour point apparatus suitable for either laboratory or production plant use, and

Figure 2 is an elevation of a preferred embodiment of the invention, particularly adapted for completely automatic operation in production plant use.

Referring to the drawings, in Figure 1, a cooling bath 10 is used as a means for cooling a sample 11 of the product to be tested. Preferably the cooling bath 10 is a conventional freezer cabinet, capable of lowering the temperature to at least the pour point of a sample to be tested, normally in the range of 20 to -50°F., however, the range may vary depending upon the characteristics of the sample. An oil or other product to be tested is introduced into a sample container 12 which is placed in position so that it may be rocked by the action of the rocking motor 13, cam 14 and rocking arm 15. The motor 13 is of fractional horsepower and the speed is reduced by means of a gear train so that the sample container 12 is rocked about once a minute. A gentle to and fro motion is preferred, however, other motions may be used, such as a revolving movement or similar. The frequency of rocking may vary, but should be such that agitation of the sample surface does not result. The cam 14 and rocking arm 15 are so selected that the amplitude of the rocking motion is preferably about 8° from the horizontal. The amplitude may also vary, and should be such as to allow a substantial deviation from the horizontal, but not so great as to allow spillage of the sample.

As the sample is cooled to the pour point a temperature sensing device 16, either immersed in the sample or preferably, placed in a thermowell in the container 12 senses the temperature of the sample. The preferred temperature sensing device is a thermocouple which is connected to a recorder 20, which records the sample temperature during the test. A source of light 17 is directed on to the surface of the sample, and the light reflected from the surface is received by a light sensitive surface 18, preferably a photoelectric cell. The reflected light rays may be received directly by the photocell 18, or mirrors may be used to pick up the light as hereinbefore described, however, it is within the scope of the invention to utilise any source of radiation or wave emanations capable of being reflected from the surface of the sample 11, together with any appropriate receiver for the reflected rays. The angle of incidence of the rays striking the surface of the sample is not critical, but is selected to conform to the physical space limitations of the cooling bath used. While the sample is liquid its surface remains substantially horizontal and the reflected light is continuously received by the photocell 18. However, when the pour point is reached the solidification causes the surface of the sample to tilt from the horizontal with the rocking motion, and the reflected light is diverted away from the photocell 18. Relays 19 are connected with the photocell 18, the rocking motor 13 and a recorder 20. The photocell current may be amplified if necessary to actuate the relays. When the reflected light fails to reach photocell 18, relays 19 are actuated and the test is concluded; the lowest temperature recorded by recorder 20 is the pour point.

In order to facilitate the introduction of sample 11 into the container 12 a sample transport motor 21 and transport arm 22 may be used to transport the container 12 from the filling position 23 to its position on the rocking arm 15 when the test is begun. Access to the fililng position may be had by opening a door 24 in the bath 10. This sample transport motor 21 may be connected with the relays 19 so that the container is transported to the filling position 23 at the conclusion of the test.

In Figure 2, wherein is shown an elevation of a preferred type of pour point apparatus, a cooling bath 26 utilises a secondary refrigerant, preferably methylene chloride, which is cooled in conventional refrigeration equipment outside the bath 26, and flows between the double walls of the bath 26. The bath 26 may be provided with a heater 27, preferably of electric resistance type, so that the cabinet may be defrosted when necessary. A fan 28 is used to circulate the air within the cabinet to obtain uniform temperatures during cooling and to speed the defrosting operation. A drain 29 is provided to remove water from the cabinet during the defrosting.

The sample 31 may be introduced into the container 32 manually by entry through door 34 and the container placed in position on the rocking arm 33. Alternately, in a completely automatic version of the pour point apparatus the sample may be introduced by means of line 36. The sample container 32 is attached to a rocking arm 33 and rocked by a rocking motor 37 and cam 38 as in the apparatus of Figure 1. A light source 39, aligned coaxially with a

photocell 41, is positioned in a plane substantially normal to the horizontal surface of the sample. The light rays pass through a clear plastic window 42, strike the surface of the sample 31 and are reflected back into photocell 41. Lens 43 serves to concentrate the emitted light and lens 43a serves to focus it after reflection from the sample surface, on the photocell 41. As in the apparatus of Figure 1, when the sample 31 is cooled to its pour point the surface tilts from the horizontal and the reflected light is diverted away from the photocell 41. This actuates relay 44, shutting off recorder 46 which has recorded the sample temperature sensed by thermocouple 47.

This apparatus is well adapted for automatically taking samples of the product to be tested directly from the production unit and testing such samples at preselected intervals of time. A timer 48 is used to regulate the functioning of all the component parts of the apparatus by individual connections or by a connection with a common power source. Any of the functional components of the apparatus may be reduced to automatic operation by the appropriate connections to the timer 48. For example, the timer 48 and driver 49 actuate valve 51 to allow a sample of the product from the production unit to pass by lines 52 and 36 into container 32. The timer 48 may also be connected to the recorder 46 and rocking motor 37 to start and stop the test automatically in conjunction with the introduction of the sample. The test is conducted as described before, and, upon being concluded, the cabinet may be heated by heater 27 to defrost the cabinet and to render the chilled sample fluid. The container 32 is drained by line 53 when valve 54 is actuated by means of the timer 48 and driver 55. The valve 56 is actuated by driver 57 connected with timer 48 to permit a flushing oil to enter the container 32 through lines 58 and 36 so as to cleanse it in preparation for receiving another sample to be tested. Valves 54 and 56 are then closed, the heater 27 is shut off and the apparatus is then ready to take another sample at the appropriate time.

A comparison was made with the Standard ASTM method by conducting several runs on a number of oils, using the standard ASTM method and the instant apparatus. The results are shown in the following table:—

TABLE I

POUR POINT (°F.)

	ASTM	*Instant Apparatus*
Oil A	—10	—11.7
Oil B	—15	—15.7
Oil C	—20	—17.5

The above data indicate that the pour points obtained by the instant apparatus are well within ± 5°F. accuracy range obtainable by the standard ASTM method.

The apparatus described with reference to Figure 2 determines the pour point of a liquid sample in a simple accurate manner, reduces the time required for a pour point test and does not require the constant attention of the operator. The apparatus is suitable for plant use and the time required by the production operator to obtain the test results is minimised. This increases the operational efficiency of a plant which produces products which must meet critical pour point specifications.

WHAT WE CLAIM IS:—

1. Apparatus for determining the pour point of a liquid sample, which apparatus comprises a container for said sample, means for cooling said sample to its pour point, means for rocking said container during the cooling of said sample so that the surface of said liquid sample remains in a substantially horizontal plane but so that said surface tilts from the horizontal when the temperature of said sample is at or below its pour point, means for detecting movement of the surface of the sample from a substantially horizontal plane, and means for indicating the temperature of said sample when the surface of said sample moves from a substantially horizontal plane.

2. Apparatus as claimed in claim 1 wherein the means for detecting movement of the surface of the sample from a substantially horizontal plane comprises a source of a beam of electromagnetic radiation and a sensitive receiver positioned so as to receive the beam after reflection from the substantially horizontal surface of the sample.

3. Apparatus as claimed in claim 1 or claim 2 wherein the electromagnetic radiation is light.

4. Apparatus as claimed in any one of the preceding claims wherein there is also provided a defroster to warm the cooling means.

5. Apparatus substantially as hereinbefore described with reference to, or as illustrated in, Figures 1 or 2 of the accompanying drawings.

6. A method of determining the pour point of a liquid sample which method comprises concurrently cooling the sample and rocking it in such a way that the surface of the liquid sample remains in a substantially horizontal plane, and then detecting the movement of the sample surface from a substantially horizontal plane and measuring the temperature of the sample at that moment.

7. A method as claimed in claim 6 wherein the movement of the sample sur-

face from a substantially horizontal plane is arranged to cause a corresponding movement of the reflection of a beam of electromagnetic radiation issuing from a stationary source and reflected from the sample surface, which movement of the beam is detected by a sensitive receiver.

8. A method as claimed in claim 6 or claim 7 wherein the amplitude and frequency of rocking are such that no substantial agitation of the surface takes place.

9. A method as claimed in claim 6 using the apparatus claimed in any one of claims 1 to 5.

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Berwick-upon-Tweed: Printed for Her Majesty's Stationery Office by The Tweeddale Press Ltd.—1964
Published at The Patent Office, 25 Southampton Buildings, London, W.C.2 from which copies may be obtained.

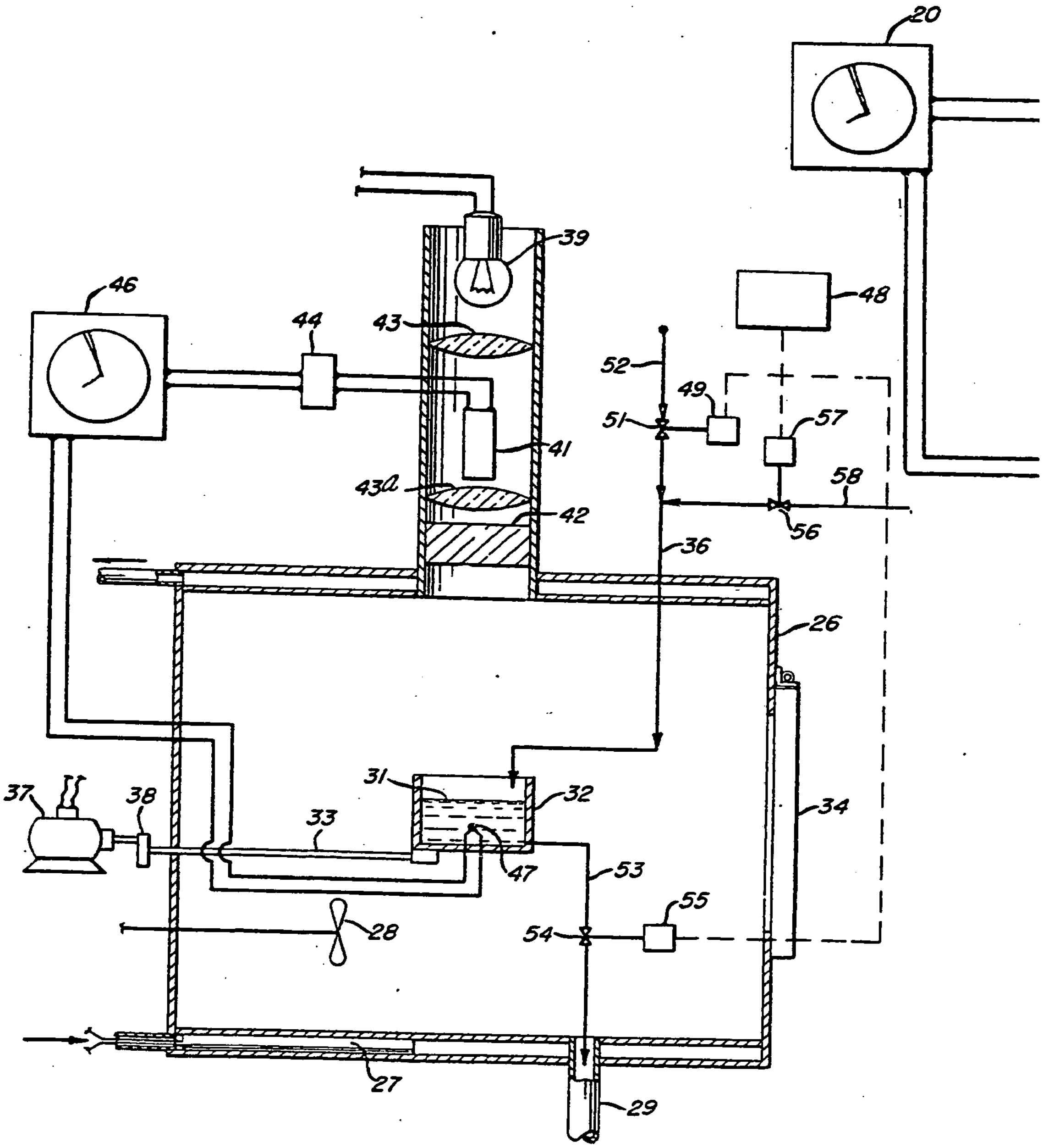
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Fig. 2

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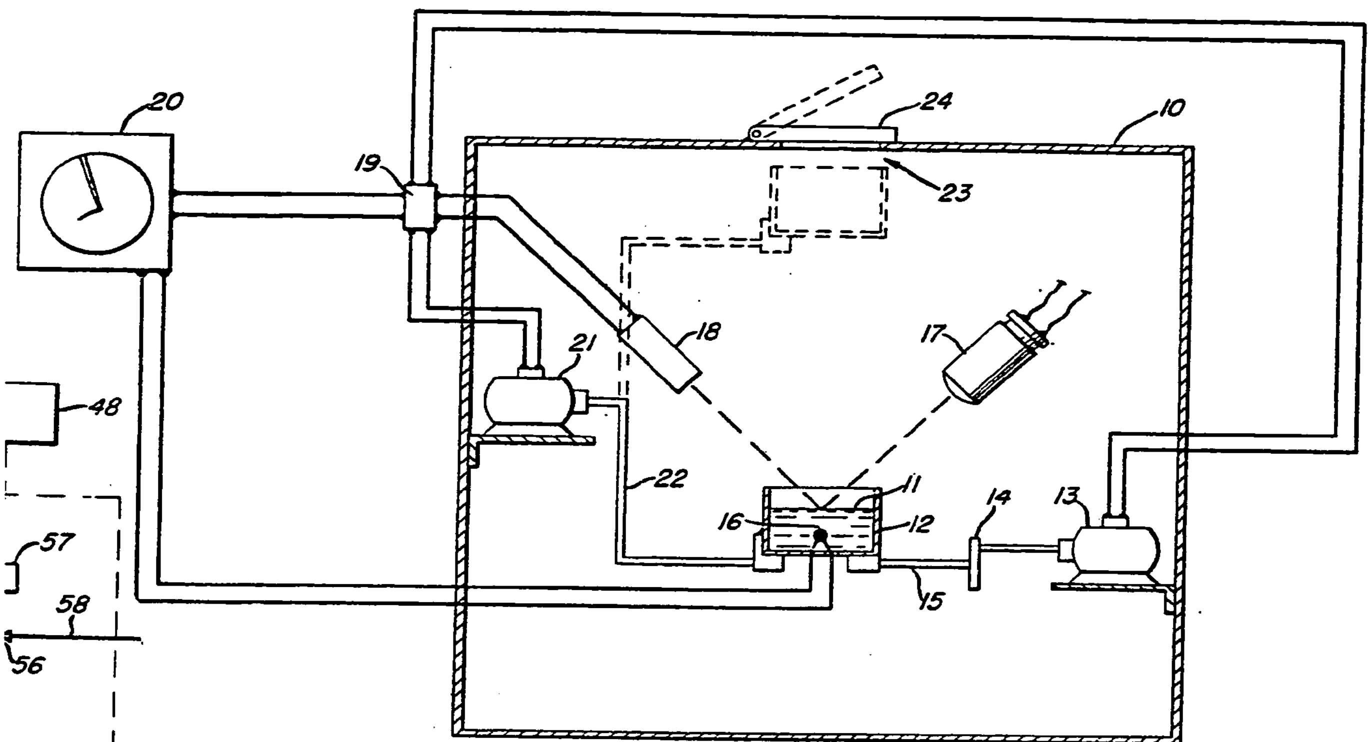

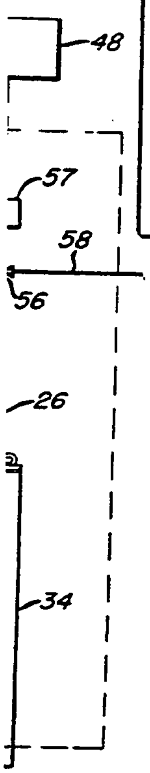

Fig. 1

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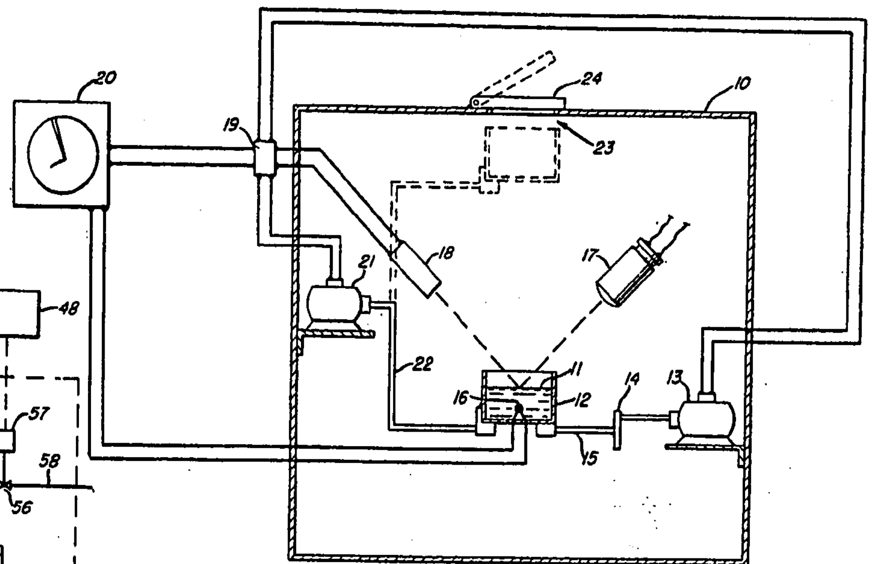

Fig. 1

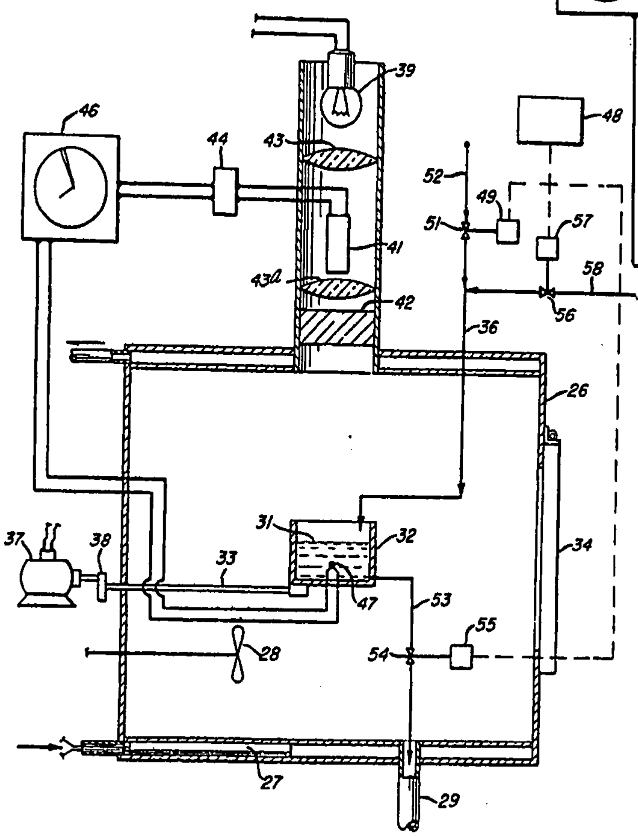

Fig. 2